

PATENT COOPERATION TREATY
PCT
INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY
(Chapter II of the Patent Cooperation Treaty)
(PCT Article 36 and Rule 70)

REC'D 20 DEC 2005

Applicant's or agent's file reference 12643PC2-MAH/JMR	FOR FURTHER ACTION	See Form PCT/IPEA/416
International application No. PCT/AU2004/001622	International filing date. (day/month/year) 22 November 2004	Priority date (day/month/year) 21 November 2003
International Patent Classification (IPC) or national classification and IPC Int. Cl. C09K 3/18 (2006.01) C09D 5/33 (2006.01) C03C 17/30 (2006.01) C09D 183/06 (2006.01)		
Applicant THE UNIVERSITY OF QUEENSLAND et al.		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 3 sheets, including this cover sheet.
3. This report is also accompanied by ANNEXES, comprising:
 - a. ☒ (sent to the applicant and to the International Bureau) a total of 4 sheets, as follows:
 - ☐ sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).
 - ☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.
 - b. ☐ (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or table related thereto, in electronic form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).
4. This report contains indications relating to the following items:

<input checked="" type="checkbox"/>	Box No. I	Basis of the report
<input type="checkbox"/>	Box No. II	Priority
<input type="checkbox"/>	Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
<input type="checkbox"/>	Box No. IV	Lack of unity of invention
<input checked="" type="checkbox"/>	Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
<input type="checkbox"/>	Box No. VI	Certain documents cited
<input type="checkbox"/>	Box No. VII	Certain defects in the international application
<input type="checkbox"/>	Box No. VIII	Certain observations on the international application

Date of submission of the demand June 2005	Date of completion of this report 8 December 2005
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au File No. (02) 6285 3929	Authorized Officer ALBERT S. J. YONG Telephone No. (02) 6283 2160

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/AU2004/001622

Box No. I

Basis of the report

1. With regard to the language, this report is based on:

☒ The international application in the language in which it was filed☐ A translation of the international application into
translation furnished for the purposes of:

, which is the language of a

☐ international search (under Rules 12.3(a) and 23.1 (b))☐ publication of the international application (under Rule 12.4(a))☐ international preliminary examination (Rules 55.2(a) and/or 55.3(a))2. With regard to the elements of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):☐ the international application as originally filed/furnished☒ the description:

pages 1-19, 24(abstract) as originally filed/furnished

pages* received by this Authority on with the letter of

pages* received by this Authority on with the letter of

☒ the claims:

pages as originally filed/furnished

pages* as amended (together with any statement) under Article 19

pages* 20-23 received by this Authority on 6 December 2005 with the letter of 6 December 2005

pages* received by this Authority on with the letter of

☒ the drawings:

pages 1/7-7/7 as originally filed/furnished

pages* received by this Authority on with the letter of

pages* received by this Authority on with the letter of

☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.3. ☐ The amendments have resulted in the cancellation of:☐ the description, pages☐ the claims, Nos.☐ the drawings, sheets/figs☐ the sequence listing (*specify*):☐ any table(s) related to the sequence listing (*specify*):4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).☐ the description, pages☐ the claims, Nos.☐ the drawings, sheets/figs☐ the sequence listing (*specify*):☐ any table(s) related to the sequence listing (*specify*):*If item 4 applies, some or all of those sheets may be marked "superseded."*

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. Statement**

Novelty (N)	Claims 1-29	YES
	Claims	NO
Inventive step (IS)	Claims 1-29	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-29	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)**NOVELTY AND INVENTIVE STEP**

Claims 1-29: The claimed invention relates to a method of forming a silica film on a substrate comprising the steps of producing a silica precursor formulation comprising silicic acid tetramethyl ester homopolymer and no more than 5% by volume of water, coating a substrate with the formulation and curing the coating in a vaporous ammoniacal environment.

None of the cited documents, or obvious combinations thereof, discloses these features. The closest art, US 6599976, discloses a composition comprising an organosilicate (MS51), a catalyst, ethanol and an excessive amount of water. Further, unlike the conventional sol-gel process, the curing step of the present invention is performed at near room temperature and atmospheric pressure. Hence, the claims are novel and inventive.

CLAIMS

1. A method of forming a silica film coated on a substrate including the steps of:

producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;

coating a substrate with the silica precursor formulation; and

curing the silica precursor formulation onto the substrate in a vaporous ammoniacal environment.
2. The method of claim 1 wherein the solvent is alcohol or an alcohol-aqueous solution.
3. The method of claim 1 wherein the silica precursor formulation contains an amount of tetramethoxysilane.
4. The method of claim 1 wherein the silica precursor formulation is formed by adding methyl-silicate-51 (MS-51), comprising >94% silicic acid tetramethyl ester homopolymer by volume, <3% tetramethoxysilane by volume and <3% methanol by volume, to the solvent.
5. The method of claim 1 wherein the silica precursor formulation comprises about 0.2-100 parts alcohol and 0.01-1 parts water for each part of MS-51.
6. The method of claim 1 wherein the silica precursor formulation comprises about 0.2 to 15 parts alcohol by volume and 0.01 to 0.1 part water by volume for each part of MS-51.
7. The method of claim 1 wherein the ratio of reagents in the silica precursor formulation is 1.0 part MS-51: 0.1 part water: 10.0 parts alcohol by volume.

8. The method of claim 1 wherein the coating is performed by spin coating or dipping.
9. The method of claim 1 wherein the coating further includes allowing the coating to settle before curing.
10. The method of claim 1 wherein the curing is carried out by placing the coated substrate in a closed ammoniacal environment.
11. The method of claim 10 wherein the ammoniacal environment contains water, ammonia and alcohol.
12. The method of claim 11 wherein the solvent used in the formation of the silica precursor is an alcohol, and the alcohol contained in the ammoniacal environment is the same alcohol as used in the formation of the silica precursor.
13. The method of claim 1 further including controlling the solvent content to control characteristics of the silica film.
14. The method of claim 1 further including controlling the alcohol content in the ammoniacal environment to control characteristics of the silica film.
15. The method of claim 1 further including controlling a pore size of the silica film by controlling the solvent content and type in the silica precursor formulation.
16. The method of claim 1 further including controlling a pore density of the silica film by controlling the solvent content and type in the ammoniacal environment.
17. The method of claim 12 further including controlling a porosity of the silica film by controlling the solvent content and type in the precursor formulation and alcohol content and type in the ammoniacal environment.

18. A silica film having a refractive index between 1.1 and 1.56 and a film thickness less than 100 microns formed by a method including the steps of:
producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;
coating a substrate with the silica precursor formulation; and
curing the silica precursor formulation onto the substrate in a vaporous ammoniacal environment.
19. The silica film of claim 18 having a thickness of less than 1 μ m.
20. The silica film of claim 18 comprising a continuous, interconnected, nanoporous silica network.
21. The silica film of claim 18 comprising a hardness greater than 7H on pencil scale.
22. The silica film of claim 18 wherein the film is resistant to washing with water, alcohols, common acids and alkalis.
23. The silica film of claim 18 wherein the film is anti-fogging.
24. Use of the silica film formed by the method of claim 1 in a coating on a transparent substrate to provide an anti-reflective and/or anti-fogging and/or protective coating
25. An anti-reflection coating for a transparent substrate comprised by a silica film formed according to the method of claim 1.
26. An anti-fogging coating for a transparent substrate comprised by a silica film formed according to the method of claim 1.
27. An anti-scratch coating for a substrate comprised by a silica film formed

according to the method of claim 1.

28. An anti-static coating for a substrate comprised by a silica film formed according to the method of claim 1.

29. A method of forming a silica film coated on a substrate including the steps of:

producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;

coating a substrate with the silica precursor formulation;

placing the coated substrate in a closed solvent environment;

establishing equilibrium between the solvent in the precursor formulation and the solvent environment; and

curing the silica precursor formulation onto the substrate in an ammoniacal environment containing solvent by introducing ammonia vapour and water vapour to the closed solvent environment.